

**Interaction of the Zirconocene Alkyne Complex  
Cp<sub>2</sub>Zr(THF)(Me<sub>3</sub>SiC<sub>2</sub>SiMe<sub>3</sub>) and the Binuclear Zirconium  
σ-Alkenyl Complex  
{Cp[μ-(η<sup>1</sup>:η<sup>5</sup>-C<sub>5</sub>H<sub>4</sub>)]Zr[C(SiMe<sub>3</sub>)=CH(SiMe<sub>3</sub>)]<sub>2</sub> with Carbon  
Dioxide and Water**

Uwe Rosenthal,<sup>\*,†</sup> Andreas Ohff,<sup>†</sup> Manfred Michalik,<sup>‡</sup> Helmar Görls,<sup>§</sup>  
Vladimir V. Burlakov,<sup>||</sup> and Vladimir B. Shur<sup>||</sup>

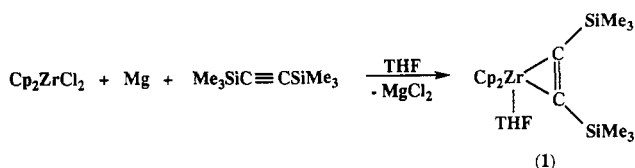
*Max-Planck-Gesellschaft, Arbeitsgruppe "Komplekxkatalyse" an der Universität Rostock,  
Buchbinderstrasse 5-6, 18055 Rostock, FRG*

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**Summary:** It has been shown that the bis(trimethylsilyl)-acetylene complex of zirconocene Cp<sub>2</sub>Zr(THF)(Me<sub>3</sub>SiC<sub>2</sub>SiMe<sub>3</sub>) (1) and the binuclear σ-alkenyl complex of zirconium {Cp[μ-(η<sup>1</sup>:η<sup>5</sup>-C<sub>5</sub>H<sub>4</sub>)]Zr[C(SiMe<sub>3</sub>)=CH(SiMe<sub>3</sub>)]<sub>2</sub> (2) readily react with carbon dioxide at room temperature to form the dimeric zirconafuranone met-

allacycle [Cp<sub>2</sub>ZrC(SiMe<sub>3</sub>)=C(SiMe<sub>3</sub>)C(O)O]<sub>2</sub> (3). Under similar conditions the reaction of Cp<sub>2</sub>Zr(THF)(Me<sub>3</sub>SiC<sub>2</sub>SiMe<sub>3</sub>) and {Cp[μ-(η<sup>1</sup>:η<sup>5</sup>-C<sub>5</sub>H<sub>4</sub>)]Zr[C(SiMe<sub>3</sub>)=CH(SiMe<sub>3</sub>)]<sub>2</sub> with water affords the zirconoxane complex [Cp<sub>2</sub>ZrC(SiMe<sub>3</sub>)=CH(SiMe<sub>3</sub>)]<sub>2</sub>O (4). The structures of the obtained complexes have been established by X-ray structure analysis.

Recently we have reported the synthesis and structure of the first alkyne complex of zirconocene, Cp<sub>2</sub>Zr(THF)(Me<sub>3</sub>SiC<sub>2</sub>SiMe<sub>3</sub>) (1), which does not contain a stabilizing phosphine ligand.<sup>1</sup> Complex 1 was obtained by the reaction of Cp<sub>2</sub>ZrCl<sub>2</sub> with equimolar amounts of magnesium and bis(trimethylsilyl)acetylene in THF at room temperature under argon.



An X-ray diffraction study of 1 has shown that the structure is close to that of a zirconacyclopropene and contains a weakly bonded THF molecule.

As a remarkable feature, 1 on dissolving in benzene or hexane at room temperature is readily transformed into the binuclear σ-alkenyl complex 2 due to hydrogen transfer from the η<sup>5</sup>-C<sub>5</sub>H<sub>5</sub> ring to the acetylene ligand.<sup>1</sup> The reaction is reversible, and dissolution of 2 in THF results in a gradual back-formation of 1. According to X-ray data and NMR spectra 2 exhibits an agostic interaction between the zirconium atoms and the alkenyl C-H bond.<sup>1</sup>

<sup>†</sup> Max-Planck-Gesellschaft, Arbeitsgruppe "Komplekxkatalyse" an der Universität Rostock.

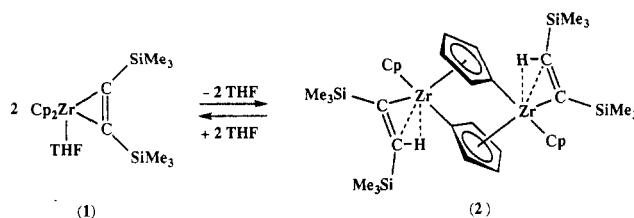
<sup>‡</sup> Institut für Organische Katalyseforschung an der Universität Rostock e.V., Rostock, FRG.

<sup>§</sup> Max-Planck-Gesellschaft, AG "CO<sub>2</sub>-Chemie" an der Universität Jena, Jena, FRG.

<sup>||</sup> Institute of Organoelement Compounds of the Russia Academy of Sciences, Moscow, Russia.

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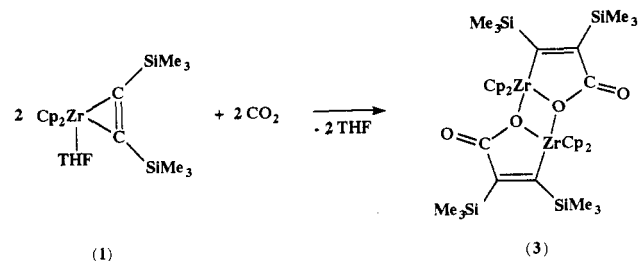
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Here we wish to report the reactions of complexes 1 and 2 with carbon dioxide and water.

### Results and Discussion

Although the interaction of carbon dioxide with titanium alkyne complexes has been well studied (see e.g. refs 2-7), similar reactions have not been described for alkyne complexes of zirconium until now. We found that 1 reacts readily with CO<sub>2</sub> at room temperature and atmospheric pressure to give the dimeric zirconafuranone metallacycle 3 in 76% yield.



Complex 3 is also formed in high yield by the reaction of 2 with carbon dioxide in THF under similar conditions. If the reaction of 2 with CO<sub>2</sub> is carried out in benzene the yield of 3 decreases.

Complex 3 is a light-yellow crystalline solid (mp 177-178 °C (dec) under argon) stable in air and practically insoluble in hydrocarbons and ethereal solvents.

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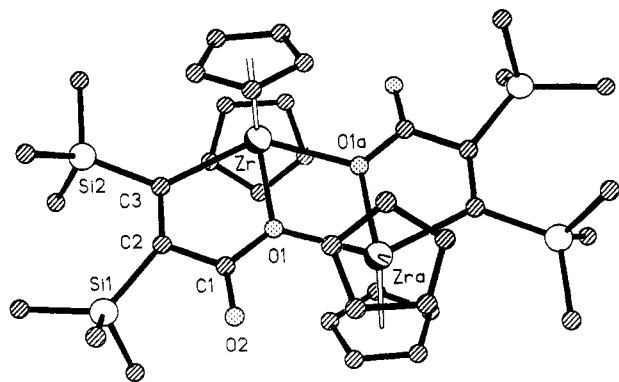


Figure 1. SHELXTL plot showing the molecular structure of 3.

Table I. Crystallographic Data for Complexes 3 and 4

	3	4
chem formula	C <sub>38</sub> H <sub>56</sub> O <sub>4</sub> Si <sub>4</sub> Zr <sub>2</sub>	C <sub>36</sub> H <sub>58</sub> OSi <sub>4</sub> Zr <sub>4</sub>
molecular weight	871.6	801.6
cryst system	triclinic	monoclinic
space group	P1̄ (No. 2)	C2/m (No. 12)
cell params at 293 K		
a (Å)	10.254(3)	17.063(2)
b (Å)	10.806(3)	12.403(2)
c (Å)	11.041(3)	10.452(1)
α (deg)	69.11(2)	
β (deg)	67.61(2)	114.96(1)
γ (deg)	66.39(2)	
V (Å <sup>3</sup> )	1006.1(5)	2005.4(5)
cryst dimens (mm)	0.40 × 0.40 × 0.16	0.40 × 0.24 × 0.20
no. of formula units (Z)	1	2
D <sub>c</sub> (g/cm <sup>3</sup> )	1.44	1.33
abs coeff μ(Mo Kα) (cm <sup>-1</sup> )	6.6	6.5
2θ range (deg)	2–55	2–55
no. of unique data	4806	4821
no. of obsd data with I ≤ 2σ(I)	4567	1940
no. of variables	217	113
R	0.042	0.033
R <sub>w</sub>	0.055	0.048
rest electron density (e Å <sup>-3</sup> )	1.47	0.43

An X-ray structure analysis of 3 revealed two zirconafuranone units linked with each other by two coordinative Zr–O bonds (Figure 1). Table I lists crystallographic data. Positional parameters and selected bond lengths and angles of 3 are given in Tables II and IV.

The molecule has C<sub>2</sub> symmetry with the symmetry center in the middle of a four-membered Zr–O–Zr–O ring. This ring is planar, and the O atoms are parts of five-membered zirconafuranone units. The zirconium atoms have a distorted trigonal-bipyramidal coordination sphere having the Cp ring centers and the O1 atom in equatorial positions and the atoms O1a and C3 in axial positions. The whole tricyclic system is almost planar. The angle of distortion of C1O1ZrO1a amounts to 178.9(2)°. The SiMe<sub>3</sub> groups are in cis positions.

Analogous titanafuranone metallacycles but having a monomeric structure were obtained previously in the reaction of carbon dioxide with titanium alkyne complexes CpCp\*Ti(PhC<sub>2</sub>Ph), Cp<sub>2</sub>Ti(HC<sub>2</sub>H)(PMe<sub>3</sub>), and Cp<sub>2</sub>Ti-

Table II. Selected Bond Lengths (Å) and Angles (deg) for 3<sup>a</sup>

Zr–O(1)	2.197(2)	C1–C2	1.488(5)
Zr–O1a	2.330(2)	Zr–CP2	2.254(3)
Zr–C3	2.376(3)	O1–C1	1.342(4)
Zr–CP1	2.243(3)	O2–C1	1.223(4)
Si1–C2	1.923(4)	C2–C3	1.360(5)
Si2–C3	1.895(4)		
Zr–O1–Zra	116.8(1)	Zr–C3–C2	114.5(2)
Zr–O1–C1	121.2(2)	Si2–C3–C2	116.8(2)
O1–C1–O2	120.4(3)	O1–Zr–O1a	63.2(1)
O1–C1–C2	116.4(3)	O1–Zr–C3	71.7(1)
Si1–C2–C3	138.4(3)	CP1–Zr–CP2	130.4(2)
C1–C2–C3	115.9(3)		

<sup>a</sup> CP1 is the centroid of the Cp-ring C10–C14, and CP2 is the centroid of the Cp-ring C15–C19.

Table III. Selected Bond Lengths (Å) and Angles (deg) for 4<sup>a</sup>

Zr–O	1.973(1)	C1–C2	1.349(6)
Zr–C1	2.299(4)	Zr–CP1	2.263(4)
Zr–C2	3.017(5)	O–C2	3.294(5)
Zr–H2	2.72(1)	O–H2	2.44(1)
Si1–C1	1.871(4)	C2–H2	1.01(1)
Si2–C2	1.874(5)		
O–Zr–C1	104.7(1)	Zr–C1–C2	108.8(3)
CP1–Zr–CP1a	128.1(1)	Si2–C2–C1	140.3(4)
Si1–C1–C2	120.3(3)	C1–C2–H2	109.6(3)

<sup>a</sup> CP1 and CP1a are the centroids of the Cp rings.

Table IV. Positional Parameters and Their Estimated Standard Deviations for 3

atom	x	y	z	B (Å <sup>2</sup> )
Zr	-0.20527(3)	0.57461(3)	0.59298(3)	2.308(6)
Si1	0.0253(1)	0.7929(1)	0.7684(1)	3.66(2)
Si2	-0.3637(1)	0.8300(1)	0.8214(1)	4.02(3)
O1	0.0253(2)	0.5673(2)	0.5481(2)	2.78(5)
O2	0.1880(3)	0.6394(3)	0.5708(3)	4.92(7)
C1	0.0600(3)	0.6397(3)	0.6015(3)	3.18(7)
C2	-0.0644(3)	0.7190(3)	0.6968(3)	2.77(7)
C3	-0.2020(3)	0.7194(3)	0.7103(3)	2.69(7)
C4	0.1208(5)	0.9138(5)	0.6297(5)	6.3(1)
C5	0.1567(5)	0.6420(6)	0.8520(5)	6.4(1)
C6	-0.0850(6)	0.8906(6)	0.9040(5)	8.4(1)
C7	-0.3620(6)	1.0154(4)	0.7492(5)	5.9(1)
C8	-0.3536(6)	0.7556(5)	1.0013(5)	6.4(1)
C9	-0.5560(6)	0.8420(6)	0.8348(6)	7.2(2)
C10	-0.4370(4)	0.7477(5)	0.5332(5)	5.0(1)
C11	-0.3782(4)	0.6568(5)	0.4493(4)	5.0(1)
C12	-0.2479(4)	0.6784(4)	0.3588(4)	4.2(1)
C13	-0.2228(4)	0.7809(4)	0.3884(4)	4.00(9)
C14	-0.3416(4)	0.8248(4)	0.4965(4)	4.3(1)
C15	-0.4018(4)	0.4896(4)	0.7925(5)	4.8(1)
C16	-0.3389(4)	0.3919(4)	0.7126(4)	4.7(1)
C17	-0.1955(4)	0.3233(4)	0.7198(4)	4.3(1)
C18	-0.1684(4)	0.3770(4)	0.8007(4)	4.2(1)
C19	-0.2951(5)	0.4785(4)	0.8464(4)	4.3(1)

(MeC<sub>2</sub>Me).<sup>2–4</sup> It is interesting that the alkyne complexes Cp<sub>2</sub>Ti(PhC<sub>2</sub>Ph) and Cp<sub>2</sub>Ti(Me<sub>3</sub>SiC<sub>2</sub>SiMe<sub>3</sub>) react with carbon dioxide differently from the above compounds. Here the reaction leads to the displacement of 0.5 mol of alkyne from the coordination sphere of titanium accompanied by formation of the binuclear titanium(III)  $\sigma$ -vinylcarboxylate derivatives Cp<sub>2</sub>TiC(R)=C(R)COOTiCp<sub>2</sub> (R = Ph, SiMe<sub>3</sub>).<sup>5</sup> The monomeric titanafuranone metallacycles are formed only in a further reaction with air oxygen.<sup>5,7</sup>

Upon reaction of 1 or 2 with water in THF at room temperature the zirconoxane complex 4 containing two  $\sigma$ -alkenyl groups is formed. Similar titanoxane  $\sigma$ -alkenyl complexes have been described.<sup>8–12</sup>

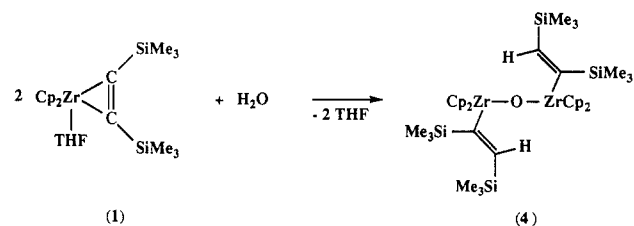
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If the reaction of 2 with water is conducted in toluene, the yield of 4 decreases as in the case of the interaction of 2 with CO<sub>2</sub> and uncharacterized byproducts are obtained.

Complex 4 is a light-yellow crystalline substance readily soluble in benzene and THF and decomposing at 202–203 °C in argon atmosphere. The structure of 4 has been determined by NMR spectroscopy and X-ray analysis (Figure 2). Positional parameters and selected bond lengths and angles are listed in Tables III and V.

A molecule of 4 has C<sub>2h</sub> symmetry in which the oxygen atom is the symmetry center. The mirror planes pass through the zirconium atoms, the carbon atoms C1 and C2, the Si atoms, and the O atom. The zirconium center has a tetrahedral coordination sphere composed of the Cp ligands, the O atom, and C1. In the alkenyl unit the SiMe<sub>3</sub> groups are in a cis arrangement.

The [Zr]–C<sub>α</sub>=C<sub>β</sub>–H unit in 4 is characterized in the <sup>1</sup>H NMR spectrum by the absorption of  $\sigma$ -alkenyl protons at  $\delta$  8.00 ppm and in the <sup>13</sup>C NMR spectrum by the large difference in C<sub>α</sub> and C<sub>β</sub> chemical shifts of the  $\sigma$ -alkenyl carbon atoms and the very low coupling constant  $J(C_{\beta}$ –H) = 87.8 Hz. Such NMR parameters are usually observed for the zirconium  $\sigma$ -alkenyl complexes with agostic C<sub>β</sub>–H...Zr interactions (see ref 13 and Table VI). However, according to the X-ray structure analysis of complex 4, the C<sub>β</sub>–H...Zr distances (2.72(1) Å) are significantly longer than those observed in 2<sup>1</sup> and in Cp<sub>2</sub>Zr(Cl){C(SiMe<sub>3</sub>)=CHPh} (5)<sup>13</sup> (Table VI) in which the existence of a C<sub>β</sub>–H...Zr agostic interaction has been established previously. The ZrC<sub>α</sub>C<sub>β</sub> bond angle in 4 (ZrC(1)C(2) angle in Figure 2) amounts to 108.8(3)°, which is significantly larger than the corresponding ZrC<sub>α</sub>C<sub>β</sub> bond angles in 2 (88.2(2)°) and 5 (89.9(2)°). On the other hand this angle is noticeably smaller than those in similar zirconium  $\sigma$ -alkenyl complexes (136.0–144.9°) in which agostic C<sub>β</sub>–H...Zr interactions were not detected.<sup>13</sup> This may indicate the possibility of the existence of weak agostic C<sub>β</sub>–H...Zr interactions in complex 4.

Another possible reason for the above-mentioned peculiarities of the NMR spectra and structure of complex 4 is the presence of weak C<sub>β</sub>–H...O hydrogen bonds in 4. The C<sub>β</sub>–H...O distance of 3.294(5) Å in 4 is in the range of such weak interactions.<sup>14</sup>

### Experimental Section

**General Considerations.** All syntheses were performed under argon using standard Schlenk techniques. THF, benzene, toluene, and hexane were distilled from sodium tetraethylaluminate under argon before use. Infrared spectra were recorded on a Nicolet Magna 550 as Nujol mulls using KBr plates. NMR measurements were done on a Bruker ARX 300. Solvents for NMR were dried over sodium and distilled under argon.

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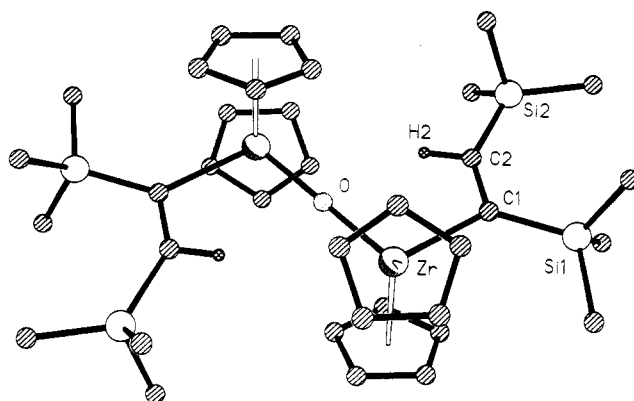


Figure 2. SHELXTL plot showing the molecular structure of 4.

Table V. Positional Parameters and Their Estimated Standard Deviations for 4

atom	x	y	z	B (Å <sup>2</sup> )
Zr	0.08027(2)	0.000	0.20196(3)	3.321(7)
O	0.000	0.000	0.000	3.65(8)
Si1	0.32548(8)	0.000	0.3552(1)	4.38(3)
Si2	0.27527(7)	0.000	-0.0312(1)	4.43(3)
C1	0.2157(2)	0.000	0.2046(4)	3.19(8)
C2	0.2072(3)	0.000	0.0703(4)	3.89(9)
C3	0.3243(4)	0.000	0.5358(6)	7.7(2)
C4	0.3842(3)	0.1248(4)	0.3500(5)	7.1(1)
C5	0.2432(3)	0.1185(6)	-0.1478(6)	11.4(2)
C6	0.3952(4)	0.000	0.0477(7)	10.7(3)
C7	0.0764(2)	0.2055(3)	0.1939(4)	5.89(9)
C8	0.0024(2)	0.1755(3)	0.1996(4)	6.17(9)
C9	0.0218(2)	0.1286(4)	0.3312(4)	7.2(1)
C10	0.1133(3)	0.1318(4)	0.4052(4)	7.6(1)
C11	0.1461(2)	0.1788(3)	0.3213(4)	6.20(9)
H2	0.144	0.000	0.003	7(1)

Table VI. Selected NMR and Structural Parameters of Complexes 4, 2, and Cp<sub>2</sub>Zr(Cl)[C(SiMe<sub>3</sub>)=CHPh] (5)

	4	2 <sup>1</sup>	5 <sup>13</sup>
<sup>1</sup> H NMR			
$\delta(C_{\beta}H)$ (ppm)	8.00	7.52/7.28	8.59
<sup>13</sup> C NMR			
$\delta(C_{\alpha})$ (ppm)	236.1	230.2/230.3	206.8
$\delta(C_{\beta})$ (ppm)	148.3	101.7/100.4	113.3
$\Delta\delta = \delta(C_{\alpha}) - \delta(C_{\beta})$ (ppm)	87.8	128.5/129.9	93.5
$^1J(C_{\beta}H)$ (Hz)	123	96/97	123
Structural Parameters			
$d(Zr-C_{\alpha})$ (Å)	2.297(4)	2.244(3)	2.258(2)
$d(Zr-C_{\beta})$ (Å)	3.017(5)	2.566(3)	2.623(2)
$d(C_{\alpha}=C_{\beta})$ (Å)	1.349(6)	1.318(5)	1.339(3)
$d(C_{\beta}-H)$ (Å)	1.01(1)	1.10(3)	0.98
$d(Zr-H)$ (Å)	2.72(1)	2.14(1)	2.29(2)
ZrC <sub>α</sub> C <sub>β</sub> (deg)	108.8(3)	88.2(2)	89.9(2)

Elemental analyses were carried out at Dornis and Kolbe, Mikroanalytisches Laboratorium, Mülheim a. d. Ruhr, FRG. Melting points were measured in sealed capillaries (1 atm of argon) on a Büchi 535 apparatus.

**Preparation of 3.** A 0.185-g amount of complex 1 was dissolved in 5 mL of THF under argon. Argon was carefully removed by evacuating and replaced by carbon dioxide, and the solution was kept at room temperature without stirring. In the course of 24 h yellow crystalline aggregates of complex 3 were slowly formed at the bottom and walls of the vessel. The solution was decanted. The remaining crystals were washed once with 2 mL of THF and dried in vacuo to give 0.132 g (76%) of 3: IR (Nujol mull) 1644 cm<sup>-1</sup> ( $\nu_{C=O}$ ); <sup>1</sup>H NMR (THF-d<sub>6</sub>)  $\delta$  0.35 (s, 18 H, SiMe<sub>3</sub>), 0.40 (s, 18 H, SiMe<sub>3</sub>), 6.09 (s, 20 H, Cp). Anal. Calcd: C, 52.36; H, 6.48; Si, 12.89; Zr, 20.39. Found: C, 52.52; H, 6.58; Si, 12.54; Zr, 20.34.

An analogous reaction of complex 2 (0.231 g) with carbon dioxide in benzene gave 3 in a yield of 0.117 g (45%).

**Preparation of 4.** A 0.734-g amount of complex 1 was dissolved in 20 mL of THF under argon, and 0.03 mL of distilled water was added to the solution. The solution immediately turned yellow. The mixture was stirred at room temperature for 2 h, filtered, and evaporated to dryness. The crude yellow product (0.551 g, 87%) was recrystallized from *n*-hexane:  $^1\text{H NMR}$  ( $\text{C}_6\text{D}_6$ )  $\delta$  0.32 (s, 18 H,  $\text{SiMe}_3$ ), 0.36 (s, 18 H,  $\text{SiMe}_3$ ), 6.01 (s, 20 H, Cp), 8.00 (s, 2 H,  $\text{C}_\beta\text{H}$ );  $^{13}\text{C}\{^1\text{H}\}$  NMR ( $\text{C}_6\text{D}_6$ )  $\delta$  2.0 (s,  $\text{SiMe}_3$ ), 3.7 (s,  $\text{SiMe}_3$ ), 111.0 (s, Cp), 148.3 (s,  $\text{C}_\beta$ ), 236.1 (s,  $\text{C}_\alpha$ ). Anal. Calcd: C, 53.94; H, 7.29. Found: C, 54.33; H, 6.89.

Alternatively, complex 4 was prepared from the reaction of 2 with water in toluene by using the procedure described above. Yield: ca. 40%, by NMR spectroscopy.

**Crystallography.** X-ray data were collected on an Enraf-Nonius CAD4 diffractometer using  $\text{Mo K}\alpha$  radiation ( $\lambda = 0.7107 \text{ \AA}$ ). Data were corrected for Lorentz and polarizations effects but not for absorption.<sup>15</sup> The structures were solved by Patterson methods<sup>16</sup> and refined by full-matrix least squares.<sup>15</sup> Hydrogen atoms were included at calculated positions with fixed thermal

parameters; non-hydrogen atoms were refined anisotropically. Cell constant and other pertinent data were collected and recorded in Table I.

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**Supplementary Material Available:** SHELXTL figures with complete atom labeling and tables of complete positional and thermal parameters, anisotropic thermal parameters, and bond distances and angles (17 pages). Ordering information is given on any current masthead page.

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